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## Procedures for Bench-Scale Ion Exchange Resin Testing for the 200-UP-1 Groundwater Operable Unit

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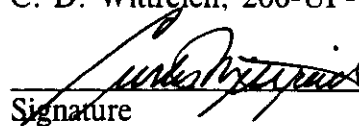
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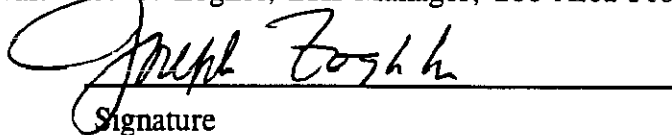
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## **PROCEDURES FOR BENCH-SCALE ION-EXCHANGE RESIN TESTING FOR THE 200-UP-1 GROUNDWATER OPERABLE UNIT**

### **1.0 INTRODUCTION**

The Pilot-Scale Treatability Test Plan for the 200-UP-1 Groundwater Operable Unit (OU), (DOE-RL 1994) identifies the need for laboratory/bench-scale testing in support of pilot-scale testing. This document describes how the laboratory/bench-scale testing will be implemented.

Laboratory testing will evaluate the effectiveness of select ion exchange resins at removing uranium and <sup>99</sup>Tc from 200-UP-1 Groundwater OU groundwater samples. Uranium and <sup>99</sup>Tc are identified as the primary contaminants of concern in the treatability test plan (DOE-RL 1994). In addition, testing will also assess resin performance for the removal of nitrate, which is a secondary contaminant.

Testing will be performed at the 200-UP-1 pilot-scale treatability test site located in the 200 West Area of the Hanford Site.

### **1.1 PURPOSE, SCOPE, AND OBJECTIVES**

The purpose of the laboratory/bench-scale testing is to obtain additional breakthrough data for the Dowex 21K (Trademark of Dow Chemical Company, Midland, Michigan) (that is correctly being used in the pilot-scale test) and to access other ion exchange resins that may be more cost-effective at removing uranium and <sup>99</sup>Tc from 200-UP-1 OU groundwater than Dowex 21K. These data, along with the results from pilot-scale testing, will be used to prepare a treatability test report. The treatability test report will support the preparation of an Interim Response Measure (IRM) Proposed Plan.

The laboratory study will consist batch equilibrium tests and flow-through column tests. Batch equilibrium tests will be performed on three resins, in addition to Dowex 21K. These tests will develop contaminant-specific loading data or isotherms for each resin tested.

Flow-through column testing will use ion-exchange mini-columns to assess the loading capacity and breakthrough characteristics of the Dowex 21K resin. These data will be used to supplement data collected under the pilot-scale test. The Dowex 21K resin will be tested at two feed rates, equivalent to contact times of 4 and 8 minutes. These tests will assess the effect of kinetics or contact time on loading capacity and breakthrough characteristics.

It is desirable that multiple loading data (i.e., run three batches of resin to breakthrough) be acquired at the pilot-scale test to adequately assess resin performance. However, only one resin breakthrough is expected during the pilot-scale test due to test limitations (e.g., 15 gal/min extraction rate, contaminant concentration, schedule). The mini-column studies described in this test plan will provide additional loading and breakthrough data on the Dowex 21K resin that is currently being used in the existing pilot-scale ion exchange process system. Breakthrough is expected between 2,000 and 4,000 bed volumes requiring approximately 50 to 100 days of pilot-scale operation. The mini-column

system can be run 24 hours/day and can treat 180 to 360 bed volumes/day. A 2,000 to 4,000 bed volume run will take 11 to 22 days.

## 2.0 EXPERIMENTAL DESIGN AND PROCEDURE

Three other resins including Lewatit MP-500-FK (trademark of Mobay Chemical company, now Miles, Inc., Pittsburgh, Pennsylvania); Rohm and Haas IRA-400 (Trademark of Rohm & Haas, Philadelphia, Pennsylvania); and Sybron Ionac A641 (Trademark of Sybron Chemical Company, Birmingham, New Jersey) will be compared to the Dowex 21K resin in batch equilibrium tests. These resins are considered to be relatively cost-effective resins for removing uranium/<sup>99</sup>Tc from the groundwater over the anticipated pH range. They are expected to demonstrate a fairly rapid adsorption of the primary contaminants and should display appropriate physical characteristics. If any of these three resins offers a significant advantage over the Dowex 21K resin, they may also be tested in the mini-column study.

The physical characteristics of different types of ion exchange resin may impact ion exchange process design. As a result, swelling potential, moisture content, and density of the different ion exchange resins will be assessed during laboratory testing.

### 2.1 BATCH EQUILIBRIUM TEST

In equilibrium distribution measurements, a known volume of groundwater is contacted with known amounts of resin and allowed to equilibrate over a 24-hour period. The resulting solution will be analyzed for uranium, <sup>99</sup>Tc, and nitrate. The four resins (Dowex 21K, Lewatit MP-500-FK, Rohm and Haas IRA-400, and Sybron Ionac A641) will each be tested at loading rates of 0.2 and 2 g of resin/L of groundwater with known <sup>99</sup>Tc, total uranium, and nitrate concentrations in duplicate. Groundwater from the pilot-scale system will provide the groundwater source for these tests. Additional batch equilibrium tests may be performed using other sources (e.g., monitoring wells) of groundwater. Procedures for batch equilibrium tests are provided in Table 1.

### 2.2 MINI-COLUMN TEST

The purpose of this mini-column study is to generate loading data at breakthrough on the Dowex 21K resin. Procedures for mini-column tests are provided in Table 2. Groundwater will be passed through separate columns at two flow rates. Two 1-in.-diameter glass columns containing a 36-in. bed of resin will be used for the test. The Dowex 21K resin will be tested at two contact times as follows:

- 4-minute contact time (134 hours to process 2,000 bed volumes)
- 8-minute contact time (267 hours to process 2,000 bed volumes).

Groundwater from the pilot-scale system will provide feed water to the columns. Resin breakthrough is expected to take place at 2,000 to 4,000 bed volumes for the primary contaminants. All columns will be run until the effluent uranium/<sup>99</sup>Tc concentration(s) is 30% to 50% of the feed concentration.

Table 1. Procedure for Batch Equilibrium Tests.

1.	Prepare the 1-L test bottles. The number of tests (i.e., bottles) to be performed is defined in Section 4.0, "Sampling and Analysis". The bottles will be labeled as follows: <ul style="list-style-type: none"> <li>• Resin name/type of Quality Control (QC) sample or feed sample</li> <li>• Resin dose</li> <li>• Replicate number.</li> </ul>
2.	Prepare duplicate tests for each resin type and for each resin dose. Measure two 0.2- and two 2-g ( $\pm 0.01$ g) quantities of each resin and place into the appropriately labeled bottles and record the weights.
3.	Place 500 to 1,000 mL of groundwater sample into each bottle including QC and feed sample bottles.
4.	Cap the bottles and place them in a tumbler for 24 hours.
5.	Remove bottles from tumbler and allow to settle for 30 to 60 minutes.
6.	Remove, as a minimum, from each bottle 20 mL for uranium analysis, 100 mL for $^{99}\text{Tc}$ analysis, and 50 mL for nitrate analysis.
7.	Measure the pH and temperature of each mixture remaining in the bottles.
8.	Record the results of all field analyses in the field notebook.

Table 2. Procedure for Mini-Column Tests.

1.	Secure ion-exchange columns in a vertical position. Attach the necessary tubing from the feed source, to the feed pump, to the test column, and then to the effluent collection tank. The feed should be at the top of the ion exchange column, with the treated effluent exiting at the bottom of the column.
2.	Place a #10 mesh screen tightly into the bottom of each ion-exchange column. Then put approximately a 2-in.-depth of the 3-mm glass beads into the column, on top of the screen.
3.	Add water, filling the column to approximately one-third of its length. Then, adding the resin through a funnel, fill to the desired column bed depth. Use a wash bottle to rinse the resin off the sides of the column. If necessary, more water can be added from the top of the column or some can be drained from the bottom. Allow the bed to settle to ensure the required bed depth is attained.
4.	Connect another pump to the bottom of the column and backwash the column with deionized water. Collect the water from the top of the column in a suitable container. The backwash rate to the column should be great enough to cause a 30% to 50% expansion of the bed volume.
5.	Continue to backwash the system until all fines are removed from the column and the effluent at the top of the column appears uncolored and clear. Then reconnect the tubing for flow from the feed tank, to the feed pump, through the top of the column, and out to the effluent storage tank.
6.	Calibrate the feed pump by measuring the flow rate of water at different pump settings. Determine the correct settings to achieve the desired flow rate for the test.
7.	Prepare appropriately sized sample bottles, as needed. The bottles will be labeled as follows: <ul style="list-style-type: none"> <li>• MCT (column number)</li> <li>• Resin name</li> <li>• Bed volume number</li> <li>• QC name and number (if applicable).</li> </ul>
8.	Start the feed water flowing through the column at the desired flow rate, and take the appropriate samples as outlined in Section 4.0, "Sampling and Analysis." <b>Important: Watch the pressure gauge at the top of the column. If the pressure rises above 5 lb/in<sup>2</sup>, shut off the feed pump and determine the cause of the pressure increase.</b> This situation may require backwashing the system or removing the top 1 to 2 in. of bed using a vacuum aspirator or other suitable device.
9.	For each sampling set, collect, as a minimum, 20 mL for uranium analysis, 100 mL for <sup>99</sup> Tc analysis, and 50 mL for nitrate analysis. Measure the pH and temperature of the water. Record the results of all field analyses in the field logbook.
10.	Once breakthrough or the test endpoint has been reached (30% to 50% of column feed concentration for uranium/ <sup>99</sup> Tc), shut the system down by turning off the feed pump and draining the column. <b>Note: The resin will contain concentrated contaminants and should be handled with care per the radiation work permit.</b>

### 3.0 EQUIPMENT AND MATERIALS

#### 3.1 BATCH EQUILIBRIUM TEST

In addition to resin samples, 1-L polyethylene bottles will be required as test containers. A tumbler will be used to agitate samples for a 24-hour period.

#### 3.2 MINI-COLUMN SYSTEM

The mini-column system will consist of two storage tanks, each with a capacity of approximately 200 gal, and the column test apparatus.

The feed water will be acquired from the existing pilot-scale system after the influent filter bank. This will provide site-specific groundwater that has passed through a 5- $\mu$ m filter. Groundwater will be pumped from the pilot-scale system into a storage tank that will serve as a feed tank for the column tests. Feed water will then be pumped through the columns using a variable-speed tubing pump. The effluent will be collected in a second storage tank before it is transferred back to the pilot-scale system.

The tubular mini-columns will have an inside diameter of 1 in., a length of 60 in., and will be constructed of heavy wall (1/8-in.-thick) glass with spherical joints on both ends. At the base of the column, a stainless steel mesh will be inserted into the column to support the resin bed. The column will first be loaded with 1 to 2 in. of 1/8-in. glass beads. The Dowex 21K resin will then be added to provide a 36-in. bed depth.

Sampling ports will be located at each of the column's inlet and outlet. A pressure gauge will be located before the column inlet to indicate any "plugging" of the system. A siphon break will also be located after the column to prevent the resin bed from draining.

### 4.0 SAMPLING AND ANALYSIS

Samples from batch equilibrium and mini-column testing will be analyzed for uranium,  $^{99}\text{Tc}$ , and nitrate. Uranium and  $^{99}\text{Tc}$  concentrations will be analyzed onsite at the 222-S Laboratory (analytical Level V) located in the 200 West Area. Samples sent to the 222-S Laboratory for analysis will be assigned a Hanford Environmental Information System number and follow chain-of-custody procedure Environmental Investigations Instruction (EII) 5.1 (BHI 1994). Uranium may also be analyzed using a laser kinetic phosphorescence analyzer in the field. Nitrate will be analyzed in the field (analytical Level II) using an ion concentration meter equipped with a nitrate-specific probe. pH and temperature measurements will also be made in the field. Any residual field screening samples will be returned to the 200-gal effluent storage tank.

#### 4.1 BATCH EQUILIBRIUM TESTING

The four resins (Dowex 21K, Lewatit MP-500-FK, Rohm and Haas IRA-400, and Sybron Ionac A641) will each be tested at loading rates of 0.2 and 2 g of resin/L of groundwater with known  $^{99}\text{Tc}$ , total uranium, and nitrate concentrations in duplicate. Aliquots of these 16 samples will then be taken at 24 hours for analysis of uranium,  $^{99}\text{Tc}$ , and nitrate concentrations. Two feed samples and a blank (a plastic jar with no resin and 500 mL of deionized water) will also be analyzed.

Following testing, loading isotherms (plot of loading capacity versus equilibrium contaminant concentration) will be generated for each resin type and corresponding contaminant.

#### 4.2 MINI-COLUMN TESTING

For each column test, a sample of the effluent will be taken every day, as a minimum. As a minimum, every third sample will be submitted for uranium,  $^{99}\text{Tc}$ , and nitrate analysis. The remainder of the samples will be archived. Once breakthrough of the first contaminant has been detected, the previous two samples will also be analyzed. The column will continue to be operated until a 30% to 50% breakthrough (effluent is 30% to 50% of column feed concentration of uranium/ $^{99}\text{Tc}$ ) has been achieved. Feed samples will also be taken from the influent feed tank after it is first filled and at each refill thereafter.

#### 4.3 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

Data quality is controlled by this document and the 200-UP-1 treatability test plan (DOE-RL 1994). Data quality objectives for laboratory testing are defined in the treatability test plan (DOE-RL 1994). Data collected under this study will be used to prepare a treatability test report for the OU that will be used to support an IRM Proposed Plan.

All field screening analyses will be performed in accordance with the instrument manufacturers' procedures for the analyte of interest. Field instruments will be calibrated following the manufacturers' recommendation and will be documented in the field logbook. Quality control samples for Levels V or II analyses will be collected, as a minimum, at the following frequency:

- One duplicate sample analysis for every 20 samples analyzed
- One blank (a plastic jar with no resin and 500 mL of groundwater) for every 20 samples analyzed.

If the test procedures cannot be followed for any reason, a nonconformance memo will be sent to the QC lead of the project. Corrective action recommendations will be provided in the memo and must be approved by the OU manager. Corrective actions will be described in the field logbook.

## 5.0 DATA MANAGEMENT

Field logbooks will be used to document the testing following field logbook procedure EII 1.5 (BHI 1994). All daily laboratory activities associated with the project will be recorded in the logbook. All data will be written into the logbook or onto standard formatted data entry sheets that are then incorporated into the logbook. In addition, a sketch of the mini-column test setup will be placed in the logbook.

All records management and reporting will follow standard quality assurance (QA)/QC protocol using the following guidelines:

- 100% verification on all numerical results: All raw data entries, transcriptions, and calculations are checked.
- Data validation through test reasonableness: Summaries of all test results are reviewed to determine the overall reasonableness of data and to determine the presence of any data that may be considered outliers.
- Use of trained personnel conducting tests: All technicians are trained in the application of standard procedures for analytical equipment operation.

## 6.0 HEALTH AND SAFETY

Laboratory testing will be performed at the 200-UP-1 pilot-scale treatability test site. As a result, health and safety requirements of the pilot-scale treatability test (DOE-RL 1994) will be followed including the site-specific radiation work permit (D-187, Rev. 1) and the hazardous waste operations permit. All field personnel working on this test will have completed the 40-Hour Hazardous Waste Site Worker Training.

## 7.0 WASTE MANAGEMENT

All waste will be managed in accordance by the 200-UP-1 Pilot-Scale Treatability Test Plan and Waste Control Plan.

## 8.0 REPORTS

A Treatability Test Report will be prepared summarizing the results of both the laboratory and pilot-scale testing.

## 9.0 REFERENCES

BHI. 1994, *Environmental Investigations Procedures*, BHI-EE-01, Vol. I, Bechtel Hanford, Inc., Richland, Washington.

DOE-RL. 1994, *Remedial Investigation/Feasibility Study Work Plan for the 200-UP-1 Groundwater Operable Unit, Hanford Site, Richland, Washington*. DOE/RL-92-76, Rev. 0, U.S. Department of Energy, Richland Operations Office, Richland, Washington.

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